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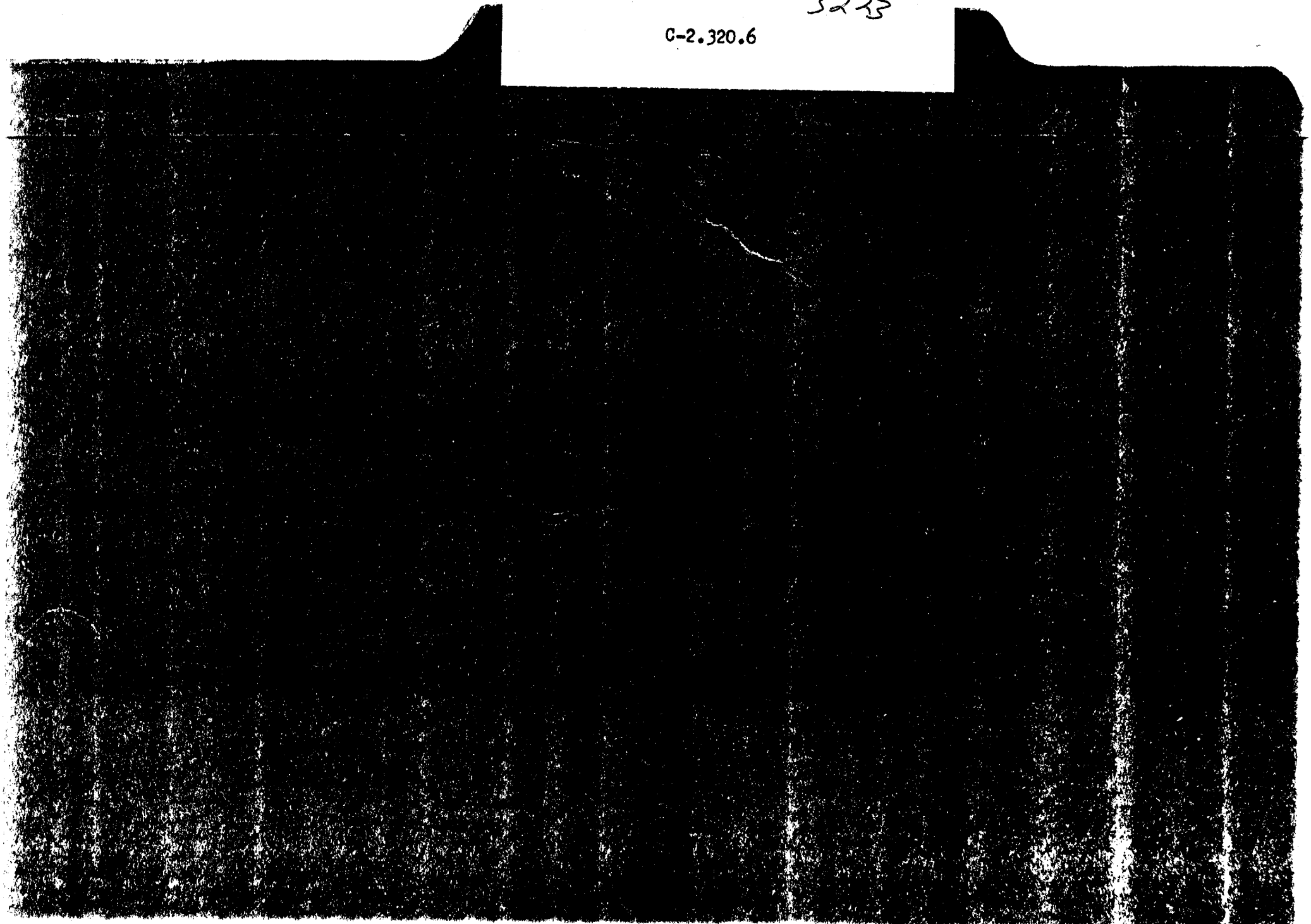

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Contract No. W-7401-eng-23

CHEMICAL DEVELOPMENT DIVISION
Dr. C. E. Larson - Director

54142

RECOVERY OF URANIUM FROM FIBERGLASS AIR FILTERS

ABSTRACT

Various procedures for extracting uranium from fiberglass air filters have been tested and compared. Satisfactory results which involve the least amount of handling are obtained by simply washing the material with hot 1:1 hydrochloric acid.

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Oak Ridge, Tennessee

August 5, 1946

SECRET

RECOVERY OF URANIUM FROM FIBERGLASS AIR FILTERS

INTRODUCTION

Previous experimental work has shown that uranium can be recovered from fiberglass air filters by dissolving the oil coating in trichlorethylene and leaching out the uranium with nitric acid. This method of treatment decreased the uranium content in the fiberglass residue from an alpha count of approximately 10,000 to approximately 100 counts per minute. Since the nicric acid leach might be considered dangerous due to possible reactions with the organic binder remaining in the fiberglass after the trichlorethylene wash, tests have been made using hydrochloric acid to leach out the uranium, with and without previous treatment with trichlorethylene.

The above tests have been checked against similar runs leached with nitric acid. Removal of the oils by ignition followed by nitric and hydrochloric acid leaches have also been made. The absolute amounts of uranium removed from the filters were determined by the alpha counting method. A count vs uranium concentration curve was established by forming standard samples from the filter constituents. This curve was found to be in excellent agreement with curve previously determined by the alpha count laboratory on standard silica and uranium mixtures.

EXPERIMENTAL METHODS AND RESULTS

Sample Preparation.--The air filter covers were removed and the fiberglass cut into twice as many pieces as samples desired. Two pieces taken from diagonally opposite parts of each half of the filter were combined to make each sample. Two samples were then ground and submitted for alpha count before treatment to determine the uranium content of the original sample.

Samples from the first air filter were treated without reducing their bulk, but samples from the second and third air filters were crushed (but not ground) to reduce the volume of solution necessary to cover them for treatment. Thus on samples from the first air filter 500 ml of trichloroethylene was used for each wash, while on the second filter 250 ml of T.C.E. was sufficient to cover each sample.

Sample Treatment.--Three filters have been sampled and the following tests run:

1. Organic removal with trichloroethylene
   a. Nitric acid leach
   b. Hydrochloric acid leach

The samples in beakers were covered with trichloroethylene and stirred for thirty minutes at room temperature. The T.C.E. was then filtered off. Samples were then covered with acid in beakers and stirred at room temperature for the cold wash. For hot washes, the acid was heated to 100°C. For reflux the samples were placed in distilling flasks, covered with acid, and refluxed under a water condenser over Ful-Kontrol heaters.

2. Organic removal by ignition (below fusion temperature of the fiberglass): The ignited samples from Filter No. 1 were heated in porcelain dishes over a burner to remove organic material. Ignitions of samples from Filter No. 3 were heated for three hours at 450°C in a muffle furnace.
   a. Nitric acid leach
   b. Hydrochloric acid leach
3. Organic removal by fusion at 650°C.
   a. Nitric acid leach
   b. Hydrochloric acid leach

4. Hydrochloric acid leach without removal of the organic material

Acids used in leachings and washings have been 1:1 dilutions of concentrated nitric and hydrochloric acids. After leaching, samples were filtered and washed on the filter paper three times with acid or water.

Analyses.--After each test run the fiberglass residue was dried at 110°C, ground to through-100 mesh, and alpha counted.

In order to determine percentage uranium variation with alpha counts, a graph was prepared, percent uranium vs alpha counts per minute, using the values for a SiO_2 background from alpha counting standards (report C-5.360.3). 1300 material was assumed present in the fiberglass and a factor of 36 was used to calculate counts for 1300 material from data reported on cold uranium. To check the predetermined SiO_2 graph, uranium in leachings from treated samples was precipitated with ammonia, the precipitate dissolved in acid, reprecipitated with ammonia, redissolved in acid, and finally precipitated with hydrogen peroxide at pH 1.8. This precipitate was ignited to U_3O_8 and ground into very low count fiberglass residues which were then recounted. These counted standards were then diluted with Berkshire sand and recounted.

No.	Residue Wt. gms.	U_3O_8 Added gms.	No.	Std. Used	Std. Wt. gms.	Sand Wt. gms.	U %	Alpha c/m
1	10.003	0.060	—	---	--	--	0.593	2752
			1a	1	5.022	5.092	0.297	1538
			1b	1	5.029	10.015	0.195	1112
			1c	---	3.064	8.017	0.170	1016
2	17.021	0.086	—	---	---	---	0.43	1836
			2a	2	5.013	5.023	0.21	1182
			2b	2	5.061	10.043	0.14	872
			2b1	2b	1.002	9.041	0.014	134
3	10.006	0.044	—	---			0.43	2360
			3a	3	0.102	50.036	0.087	610

The data obtained is plotted on Chart A in comparison with the values calculated for 1300 material in the presence of silica. The curve obtained was further checked by counting a filter sample, treating and analyzing the filtrate, and recounting the residue.

Ignited sample count	4120 c/m
Count after nitric leach	149 c/m
Sample weight	4.103 gm
Uranium in leachings (by analysis)	36.75 mg
Percentage uranium removed (by analysis)	0.896
(from graph)	0.945
Deviation of graph result from analysis	5%

Test Data

The following data sheet presents tests run and results obtained. Removal of the organic material by ignition decreased the sample weights by 15-20 percent and increased the alpha counts by 20-25 percent on untreated samples. Organic materials present in original samples evidently absorb alpha particles, thus accounting for the excess increase in count after ignition had removed organics. Trichloroethylene removed 8-12 percent of the sample weights but left discolored residues which may have counted lower than the same samples would have counted with organic matter completely removed. The decrease in sample weights on ignition has been taken into account in the data columns listing the uranium in residue from a 400 gram filter and percent uranium recovered.

All uranium percentages have been obtained from Chart A, the graph of alpha counts per minute vs percentage uraniur

The ignition sample from Filter No. 1 which was refluxed with nitric acid was evidently partially fused, thus accounting for the high count of 1368 c/m.

To determine the feasibility of successive leaches, samples were ignited and leached three times with 1:1 hydrochloric acid after ignition, with the following results:

	Sample 1	Sample 2
Ignited counts	5848	6792
Count after first leach	190	203
Count after second leach	124	124
Count after third leach	102	107

The trichloroethylene wash removed some uranium as evidenced by the following test. Berkshire sand was counted and then stirred with 250 ml of T.C.E. wash for 30 minutes, then recounted.

Count before treatment with T.C.E.	4.5 c/m
Count after stirring with T.C.E.	1674 c/m

DISCUSSION

Uranium can be removed from fiberglass air filters with hydrochloric acid to the same extent as with nitric acid leaching both in the case where previous treatment was given with trichlorethylene and where it was not. Trichlorethylene aids in removal of the uranium by dissolving the oil filter coating, but would require further treatment to recover the small amount of uranium dissolved in the trichlorethylene. Refluxing of the fiberglass with acid decreases the uranium content of the residue to a greater extent than washing. Hot washes dissolve considerably more uranium than cold washes.

Removal of the organic material from the fiberglass by ignition below the melting point of the fiberglass effectively removes the organic matter and leaves the uranium in a form readily dissolved. If the temperature is uncontrolled and the fiberglass becomes fused, the leaching processes will be ineffective in uranium removal.

Various combinations of the above steps may be made which will constitute a satisfactory plant process. The simplest method would incur crushing the fiberglass, stirring the solid with 2.5 gallons of hot 1:1 HCl, filtering and washing with 1.5 gallon of water. This method would probably result in recovery of about 99% of the uranium from a 400 gram filter contaminated with approximately 4 grams--leaving about 40 mg or 100 ppm in the residue.

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J. A. Rogers

K. B. Brown

Approved: A. J. Miller

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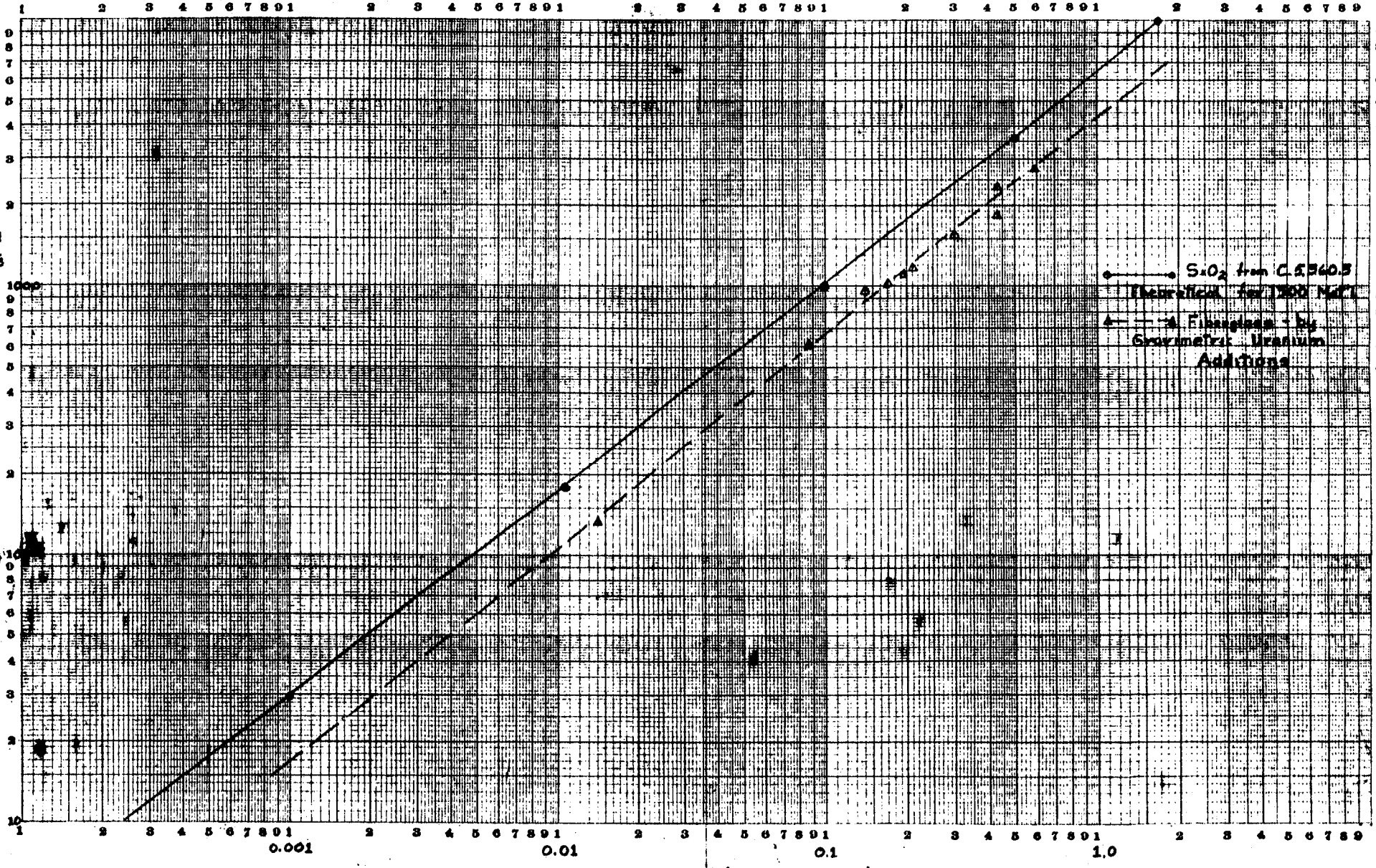
37. Mr. K. B. Brown

DATA SHEET

Filter No.	Count	Sample Wt. gms	Organic Removal	Acid Treatment			Wash		Total Treatment vol. ml	Treatment Volume per 400 gm Filter liters	Residue c/m	% U in Residue	U in Residue per 400 gm Filters mgs	% U Recovered
				Acid	Type	Vol. mls	Type	Vol. mls						
1	7160	42	T.C.E.	HNO_3	Reflux	1000	HNO_3	500	2000	19.0	17.5	0.001	4	99.92
	7160	42	T.C.E.	HNO_3	Hot Wash	1000	HNO_3	500	2000	19.0	137	0.014	56	98.88
	7160	42	T.C.E.	HNO_3	Cold Wash	1000	HNO_3	500	2000	19.0	219	0.025	100	98.00
	7160	41	T.C.E.	HCl	Reflux	1000	HCl	1000	2500	24.4	105	0.010	40	99.20
	7160	42	T.C.E.	HCl	Hot Wash	1000	HCl	1000	2500	23.8	119	0.012	47	99.06
	7160	42	T.C.E.	HCl	Cold Wash	1000	HCl	1000	2500	23.8	234	0.027	108	97.84
	7160	45	Ignition	HNO_3	Reflux	50	none	1000	50	0.4	1368	0.240	816	83.68
	7160	41	Ignition	HCl	Reflux	200	HCl	200	400	3.9	306	0.037	126	97.48
	7160	40	none	HCl	Reflux	1000	HCl	1000	2000	20.0	318	0.040	160	96.80
	7160	40	none	HCl	Reflux	1000	HCl	1000	2000	20.0	275	0.033	132	97.36
2	4400	35	T.C.E.	HNO_3	Reflux	250	H_2O	150	650	7.4	10.5	0.0005	2	99.93
	4400	35	T.C.E.	HNO_3	Hot Wash	250	H_2O	150	650	7.4	17.5	0.0010	4	99.87
	4400	35	T.C.E.	HNO_3	Cold Wash	250	H_2O	150	650	7.4	60.0	0.0048	19	99.37
	4400	35	T.C.E.	HCl	Reflux	250	H_2O	150	650	7.4	18	0.0011	4	99.87
	4400	35	T.C.E.	HCl	Hot Wash	250	H_2O	150	650	7.4	120	0.0117	47	98.43
	4400	35	T.C.E.	HCl	Cold Wash	250	H_2O	150	650	7.4	139	0.014	56	98.13
	4400	35	Fusion	HNO_3	Reflux	250	H_2O	150	400	4.6	2752	0.59	2006	33.13
	4400	35	Fusion	HNO_3	Hot Wash	250	H_2O	150	400	4.6	2868	0.61	2074	30.87
	4400	35	Fusion	HNO_3	Cold Wash	250	H_2O	150	400	4.6	2748	0.59	2005	33.17
	4400	35	Fusion	HCl	Reflux	250	H_2O	150	400	4.6	2034	0.40	1360	54.67
	4400	35	Fusion	HCl	Hot Wash	250	H_2O	150	400	4.6	2542	0.54	1836	38.80
	4400	35	Fusion	HCl	Cold Wash	250	H_2O	150	400	4.6	2748	0.59	2005	33.17
3	5000	10	none	HCl	Reflux	250	H_2O	150	400	16.0	32.5	0.0022	9	99.78
	5000	10	none	HCl	Reflux	250	H_2O	150	400	16.0	71.5	0.0062	25	99.38
	5000	10	none	HCl	Cold Wash	250	H_2O	150	400	16.0	146.5	0.015	60	98.50
	5000	10	none	HCl	Cold Wash	250	H_2O	150	400	16.0	153.5	0.016	64	98.40
	5000	10	Ignition	HCl	Reflux	250	H_2O	150	400	16.0	59.5	0.0047	16	99.60
	5000	10	Ignition	HCl	Reflux	250	H_2O	150	400	16.0	71.0	0.0060	20	99.50
	5000	10	Ignition	HCl	Cold Wash	250	H_2O	150	400	16.0	84.0	0.0074	25	99.38
	5000	10	Ignition	HCl	Cold Wash	250	H_2O	150	400	16.0	58.5	0.0046	16	99.60

CHART A

PERCENTAGE URANIUM IN FIBERGLASS



7-30-46 R.H.A.